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THERMAL

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SHOCK METHOD

Dale P. Hilleary  
James M. Lightfoot

APRIL 1989

Process Development  
Advanced Manufacturing Development  
(AMP-1)

DEPARTMENT OF ENERGY DECLASSIFICATION REVIEW	
1ST REVIEW DATE: 9-16-93	1. DETERMINATION (CIRCLE NUMBER(S))
AUTHORITY: DACC DACC ADD	1. CLASSIFICATION RETAINED
NAME: C.P. DEMOS	2. CLASSIFICATION CHANGED TO: _____
2ND REVIEW DATE: 2-25-94	3. CONTAINS NO DOE CLASSIFIED INFO
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NAME: J.R.L.	5. CLASSIFICATION CANCELLED
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## W48 DISASSEMBLY USING A THERMAL SHOCK METHOD

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### ABSTRACT

This report discusses a thermal shock method developed to remove high explosives (HE) from around the pit, detonators, and detonator cables in the W48. The method alternately heats and cools the unit using liquid nitrogen and water until the HE cracks and comes off.

The results show that the pit is freed by the thermal shock method. The results also show that it is a safe procedure that does not change the density or composition of the HE.

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## INTRODUCTION

To disassemble the W48, the PBX 9404 high explosive (HE) must be removed from the pit. In the past, Pantex has used a hand-powered cutting method. This method sawed most of the HE from the pit, the rest was dissolved using a 50/50 mixture of dimethylformamide (DMF) and acetone. The safety of this method is questionable when one considers that operator-attended cutting of PBX 9404 is not allowed in normal explosive machining, but was being done in a nuclear explosive operation. Therefore, an alternate procedure had to be found.

An interim method used was to soak the entire assembly in DMF/acetone. This procedure was time consuming, used significant quantities of flammable and toxic solvents, generated larger quantities of hazardous wastes, and did not allow retrieval of composition or density data required by the design lab. Therefore, the thermal shock disassembly procedure was developed.

In the thermal shock procedure, the HE and pit were subjected, alternately, to hot water and liquid nitrogen (LN<sub>2</sub>). Ultimately, the HE would crack and could be removed by hand. The thermal shock shortened the time for HE removal, used less solvent, and yielded the data required by the design lab.

This report discusses the procedure, temperature analysis, and density and composition of the HE after thermal shock. It also discusses the additional design agency requirements to save the detonators and detonator cables.

## DISCUSSION

Initially only eight units were thermally shocked; however, additional requirements, added by the design agency, expanded the number of tests. Some of these requirements included measuring the temperature of the pit using thermocouples, measuring the density and composition of the HE after thermal shock, and removing the detonators and detonator cables.

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The PBX 9404 charges were pressed to a W48 program nominal density of [redacted]. The adhesive used for bonding the charges to the pit was the same as initially used in the W48 program [redacted].

## INITIAL THERMAL SHOCK TESTS

The initial tests were conducted to determine the relative effects of different thermal conditions and/or sequences on the degree of HE cracking and ease of removal. Figure 1 shows the W48 configuration as tested and Figure 2 shows how the unit was submerged. The test conditions and results are summarized in Table I.

Due to the availability of magnesium caps, aluminum (simulator) caps were used for the first five tests.

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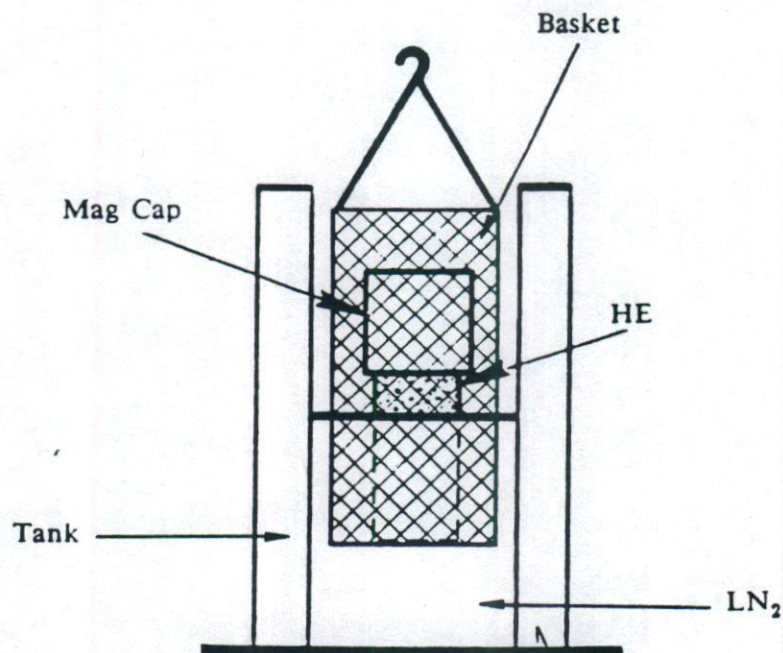


Figure 2. W48 Unit in LN<sub>2</sub> Tank

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Table I. Initial Thermal Shock Tests

Unit No.	Operation	Temperature (°F)	Time	Comments	Results
1	Conditioned in oven	180	8 hrs.	Oven was preheated	The equator was cracked, but the unit remained intact. Since this method did not work, we decided to use the same unit and try again.
	Immersed in LN <sub>2</sub>	-320	2 - 4 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	
	Maintained	ambient air	10 mins.		
	Immersed in LN <sub>2</sub>	-320	2 - 4 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	
*	Immersed in LN <sub>2</sub>	-320	10 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	The PBX 9404 crumbled, freeing the pit. The pit was cleaned by soaking it in DMF/acetone for 30 minutes.
	Submerged in water	187	5 mins.	Totally submerged, aft end first	
	Immersed in LN <sub>2</sub>	-320	5 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	
	Submerged in water	175	40 mins.	Totally submerged, aft end first	
2	Submerged in water	200	10 mins.	Totally submerged, aft end first	The HE broke into two pieces, freeing the pit. The pit was cleaned by soaking it in DMF/acetone for 30 minutes.
	Immersed in LN <sub>2</sub>	-320	10 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	
	Immersed in water	174	28 mins.	Totally submerged, aft end first	
3	Conditioned in oven	182	8 hrs.	Oven preheated	The HE crumbled and freed the pit. The remaining HE was removed from the pit by hand in about 35 minutes. The pit was cleaned by soaking it in DMF/acetone for 30 minutes.
	Immersed in LN <sub>2</sub>	-320	10 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	
	Maintained	ambient air	20 mins.		

\*Same unit as the first; second try

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Table I. Initial Thermal Shock Tests (Cont'd.)

Unit No.	Operation	Temperature (°F)	Time	Comments	Results
4	Immersed in LN <sub>2</sub>	-320	20 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	The HE crumbled and the pit was freed. Final cleaning of the pit was accomplished by soaking it in DMF/acetone for 30 minutes.
	Submerged in water	196	54 mins.	Totally submerged, aft end first	
5	Immersed in LN <sub>2</sub>	-320	12 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below aluminum cap	When the unit was removed from the water, it had small cracks in it. In 2 hours, both ends of the HE fell off the pit. The remaining HE was removed by hand in 20 minutes and the pit was cleaned by soaking it in DMF/acetone for 30 minutes.
	Submerged in water	ambient	21 mins.	Totally submerged, aft end first	
6	Immersed in LN <sub>2</sub>	-320	15 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap	The HE crumbled from the pit in 8 hours. The remaining HE was cleaned off by soaking it in DMF/acetone for 30 minutes. We later found a poor adhesive bond that may have contributed to the ease of HE removal.
	Placed on HE cart	ambient air	8 hrs.		

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Table I. Initial Thermal Shock Tests (Cont'd.)

Unit No.	Operation	Temperature (°F)	Time	Comments	Results
7	Immersed in LN <sub>2</sub>	-320	30 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap	The unit's HE developed cracks around the mag cap, but we were unable to remove the pit from the HE.
	Maintained	ambient air	23.5 hrs.		
8	Submerged in dry ice	-109	8 hrs.	Totally submerged, aft end first	The unit developed small cracks, but we were unable to free the pit from the HE.
	Placed in HE cart	ambient air	16 hrs.		

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These tests showed that some thermal shock methods can be used to remove HE from a W48 pit. The method used on Unit 4 (or slight variation thereof) was chosen for the remaining tests because of its simplicity and effectiveness.

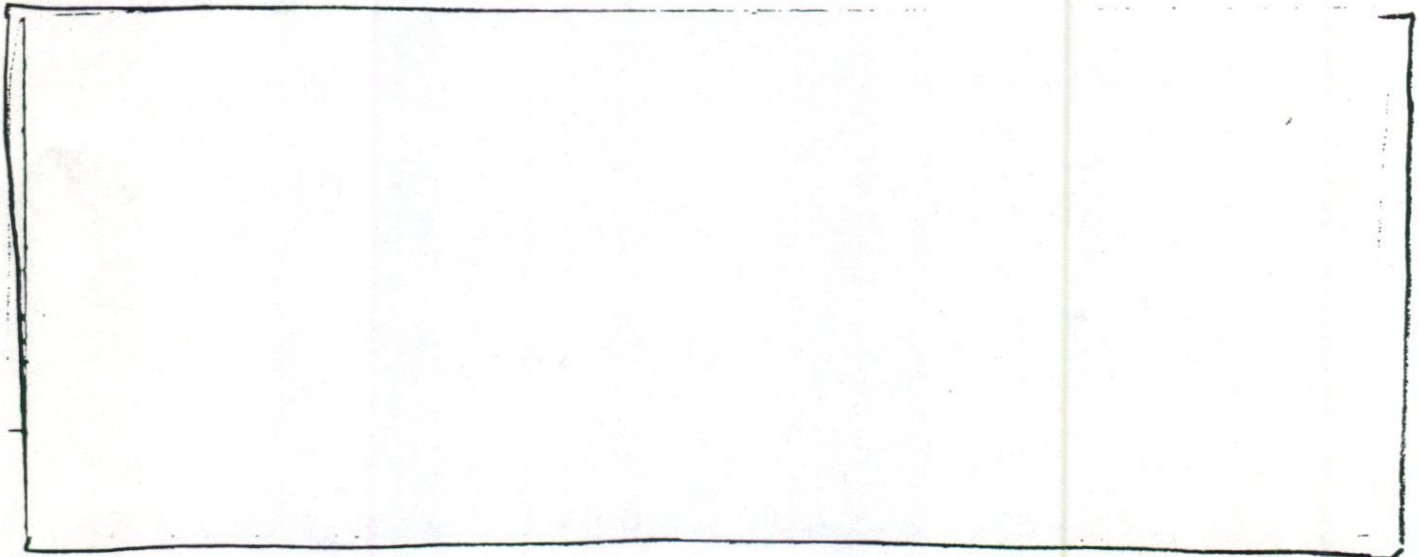
#### MEASURING TEMPERATURES WITHIN THE UNIT DURING THERMAL SHOCK

Since the thermal shock method involved the sequential immersion of explosive casings in  $LN_2$  followed by hot water or steam, concern about possible damage to the pit arose.

The units were instrumented with thermocouples to measure the temperatures within the unit during the thermal shock procedure (Figure 3). Eight thermocouples were used.

[REDACTED] The temperature versus time graphs for each test are presented in Appendix A.

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The four tests were conducted as described in Table II.

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Table II. Thermal Shock Tests to Obtain Temperature Data

Unit No.	Operation	Temperature (°F)	Time	Comments	Results
1	Immersed in LN <sub>2</sub>	-320	30 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap	Both ends broke off within 1 hour from immersion in LN <sub>2</sub> . This left a strip of explosive about 8 inches wide around the equator of the unit. The remaining explosive was removed in about 4 hours by soaking in it DMF/acetone.
	Submerged in water	185	12 mins.	Totally submerged, aft end first	
	Maintained	Ambient air	18 mins.		
2	Immersed in LN <sub>2</sub>	-320	30 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap	Small cracks were observed on both ends, but the unit was still intact.
	Sprayed with steam	212	10 mins.	Steam was at 5 psi	
	Maintained	Ambient air	16 hrs.		
3	Immersed in LN <sub>2</sub>	-320	10 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap	Both ends broke off within 1 hour and 11 minutes from immersion in the LN <sub>2</sub> . The remaining explosive was removed in about 4 hours by soaking it in DMF/acetone.
	Subjected to hand-poured water	205	10 mins.	Placed on a support to keep it from standing in water during the pouring operation	
4	Maintained	Ambient air	51 mins.		Both ends broke off within 50 minutes. The remaining explosive was removed in about 4 hours by soaking it in DMF/acetone.

\*Test conditions same as Unit No. 3

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During these tests, the temperature never became severe enough to damage the pit. We sent our data to LLNL for further analyses. They sent a letter back saying, "Nothing to date indicates the weld will fail as a result of the thermal shock process. In addition, most of the development thermal shock tests have been done [redacted]"

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LLNL also requested density and composition information on the HE. Unfortunately, the HE was cracking into very fine pieces, making it difficult to get good density measurements. We decided to run separate tests to gather this information.

#### DENSITY AND COMPOSITIONAL STUDY OF HE AFTER THERMAL SHOCK

The density and compositional data from HE in stockpiled W48 units is gathered every year. At LLNL's request, we conducted a study to determine whether the thermal shock method would change the density and/or the composition in these units.

Eight simulated [redacted] W48 units underwent thermal shock treatment and were analyzed for changes in density and composition as a result of the disassembly process.

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Test conditions for the eight units follow in Table III.

1. Letter from W. H. Hubbell, Lawrence Livermore National Laboratory, to D. W. Garrett, Mason & Hanger - Silas Mason Co., Inc. (August 28, 1987).

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Table III. Thermal Shock Tests to Obtain Density and Compositional Data

Unit No.	Operation	Temperature (°F)	Time	Comments	Results
1,2,3	Immersed in LN <sub>2</sub>	-320	10 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap, mag cap below top of LN <sub>2</sub> container (Figure 4)	The cores removed had cracks, but appeared to be suitable, for determining density and composition.
	Subjected to hand-poured water	205	10 mins.		

Note: In an effort to reduce cracks under the mag cap, the level of LN<sub>2</sub> was raised to the top of the container (Figure 5). The mag cap was 0.5 inch above LN<sub>2</sub>. By doing this, the HE and mag cap were exposed to less extremes in temperature and produced fewer cracks. Units 4 through 8 were tested in this manner.

4,5,6,7	Immersed in LN <sub>2</sub>	-320	10 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap	The cores had fewer cracks and were analyzed for density and composition.
	Subjected to hand-poured water	205	10 mins		

Note: LLNL requested that we take more thermocouple data and send it to them for stress analysis. Temperature versus time graphs for Unit 8 are presented in Appendix B.

8	Five thermocouples were attached to outside of the unit (Figure 6)	Locations for thermocouples were: 1. Top center of the mag cap 2. 0.5 inch from the bottom of the mag cap 3. 0.5 inch below mag cap on the HE surface			
	Immersed in LN <sub>2</sub>	-320	10 mins.	Aft end first, LN <sub>2</sub> 0.5 inch below mag cap	Seven analytical cores were machined from the forward charge of each unit in the typical fashion used in stockpile surveillance coring. In addition, a second set of cores was machined one layer further into the forward charge.
	Subjected to hand-poured water	205	10 mins.		
	Cores were removed for analysis				

Discussed in text below.

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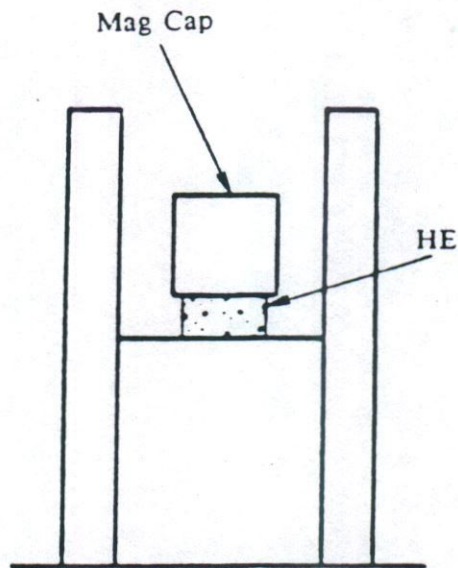


Figure 4. Initial Container Configuration

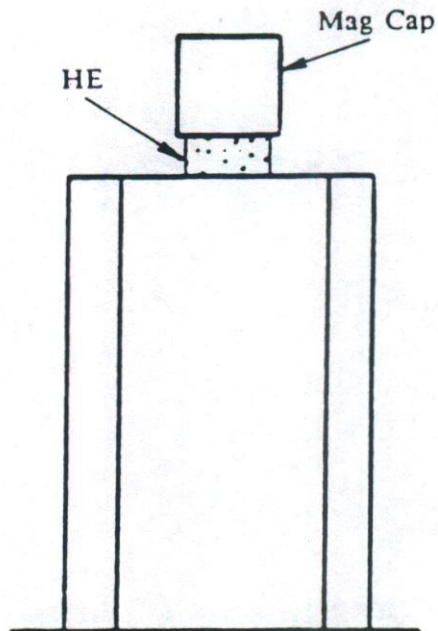


Figure 5. Final Container Configuration

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LLNL required that the [redacted] not go below 0°F. The thermocouple in that area (No. 1) experienced temperatures from 70 to 120°F, which met their requirement.

All cores were analyzed by the Quality Chemistry Laboratory.

#### Analytical Core Density Determinations for Thermally Shocked PBX 9404

The eight simulated W48 units that underwent thermal shock treatment were analyzed for changes in density as a result of the disassembly process. The first and second sets of cores, described in Table III, were identified as top and bottom, respectively. A set of cores from a W48 pressing that had not undergone thermal shock was used as a control in the evaluation. Hydrostatic density determinations were performed for the 17 sets of cores, and the average value for each set is shown in Table IV.

Table IV. Average Core Density

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<u>Unit No.</u>	<u>Mag Cap Serial No.</u>	<u>Top</u>	<u>Bottom</u>	<u>Average</u>
Control				
1	49356*			
2	63704*			
3	63078			
4	63992			
5	63650			
6	49663			
7	49356*			
8	63704*			
Average				

\*Indicates a mag cap serial number which was used twice in the experiment.

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Analysis of variance was applied to the data and significant differences between the units and between the top and bottom sets exist at the 99% level. Hydrostatic density measurements have an uncertainty of about  $0.3 \text{ mg/cm}^3$  and even though there was a statistical difference, the differences were very slight and considered to be negligible.

#### Chemical Analyses of Thermally Shocked PBX 9404

Since HMX, RDX, and nitrated diphenylamine particles are small relative to the width of the thermal gradient, which moves through the PBX when it is exposed to  $\text{LN}_2$ , quantitative analyses for these compounds were not performed. Binder analyses were performed since the polymers are of considerable length. Compositional analyses for chloroethyl phosphate, nitrocellulose, and nitrogen in nitrocellulose were performed for the first three thermally shocked units versus the W48 pressing (control). The results are presented in Table V.

Table V. Binder Compositional Analysis for the First Three Simulated W48 Units and Control

Unit No.	Percentage CEF	Percentage NC	Percentage N in NC
Control	2.71	3.37	10.50
1	2.72	3.29	10.63
2	2.69	3.20	10.76
3	2.77	3.17	10.91
Measurement Uncertainty	0.23	0.19	0.20

CEF = Chloroethyl Phosphate  
NC = Nitrocellulose  
N = Nitrogen

Analysis of variance was applied to each analysis and no significant difference was found between the units and the control at the 95% level.

Molecular weight determinations of nitrocellulose were also performed for these units and the control (Table VI). Sampling of the stockpile for this analysis is unique in that one sample is taken from a central location and another is obtained near the periphery of the aft charge. The samples used in this analysis were from the forward charge analytical cores.

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Table VI. Molecular Weight Analysis for Nitrocellulose in the First Three Simulated W48 Units and the Control\*

Unit No.	$M_w$	$M_n$
Control #1	14120	3413
Control #2	14018	3466
1	13418	3481
2	12488	3344
3	12579	3398
Measurement Uncertainty	983	155

\*Values are based on polystyrene calibration standards.

$M_w$  = Weight average molecular weight  
 $M_n$  = Number average molecular weight

ANOVA results showed a significant difference in the  $M_w$  values at the 99% level without a significant difference in the  $M_n$  values at the 95% level. Further calculations involving Tukey's Honest Significant Difference (HSD) show units 63078 and 63704 were different from the two control samples. A change in  $M_w$  values without a coincident change in  $M_n$  values indicates that the longer molecular chains were broken after being subjected to thermal shock.

The next five simulated W48 units were thermally shocked but under a less severe process. These units were evaluated for molecular weight changes in an identical fashion as above, but with only one control sample as shown in Table VII.

Table VII. Molecular Weight Analysis for Nitrocellulose in the Last Five Simulated W48 Units and the Control

Unit No.	$M_w$	$M_n$
Control	14522	3622
5	14664	3590
6	14350	3634
7	14810	3634
8	14513	3575

The results are very close and no statistical difference was observed.

In summary, the thermal shock method has negligible effects on the density, chemical composition, and molecular weight of PBX 9404.

During these tests, LLNL requested that we also try to save the detonators and their cables while the unit was going through thermal shock. We elected to wait until the process was transferred to the Pantex Production area before trying this.

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## THERMAL SHOCK TOOLING TRYOUT FOR PRODUCTION

The thermal shock method was ready for transfer from development to production. Three simulated assemblies, containing PBX 9404 explosive, were sent to production to check out the thermal shock process and tooling. One unit had thermocouples and strain gages bonded to the pit to gather strain data. These data were intended to reveal if the pit was in danger of cracking during the thermal shock process. At the request of LLNL, we would also try to save the detonators and detonator cables by means of a protective cap (Figure 7).

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This procedure also uses less time (4 hours versus 48 hours) and less material (10 gallons versus 100 gallons) than the previous test dissolution method.

The thermal shock method has been transferred from development to production. In May 1968, Nuclear Safety approved the method and in June 1968, the first production unit was disassembled.

### ACKNOWLEDGMENT

The authors would like to thank Larry R. Henschel for his help in transferring the thermal shock method to production.

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The pit did not crack, we were able to protect the detonators and detonator cables from 0°F temperatures, and the transfer to production tooling was successful. The strain gage results satisfied LLNL that the pit was safe from thermal shock damage.

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## APPENDIX A

### Unit Temperatures During Thermal Shock

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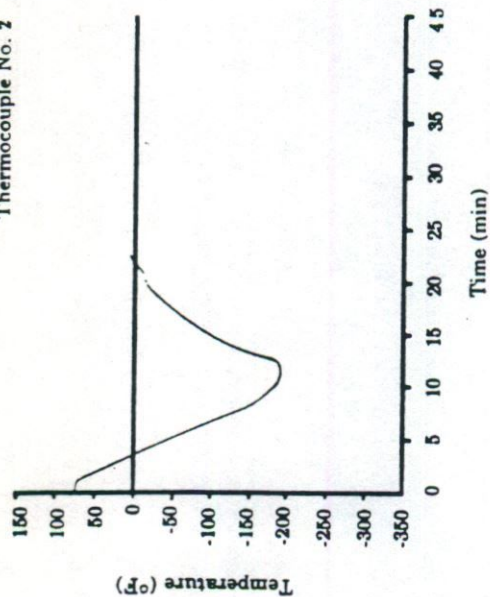
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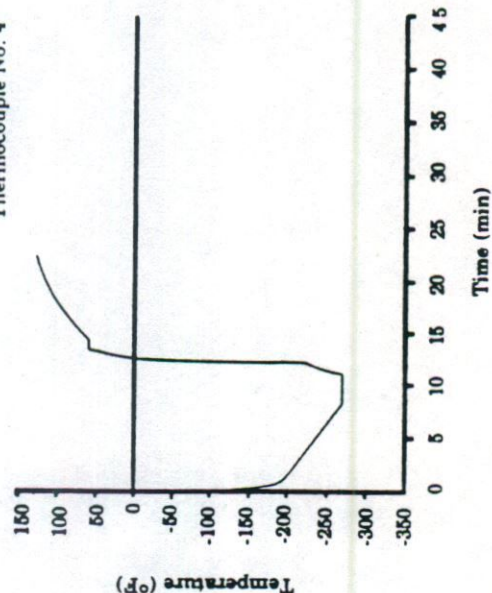


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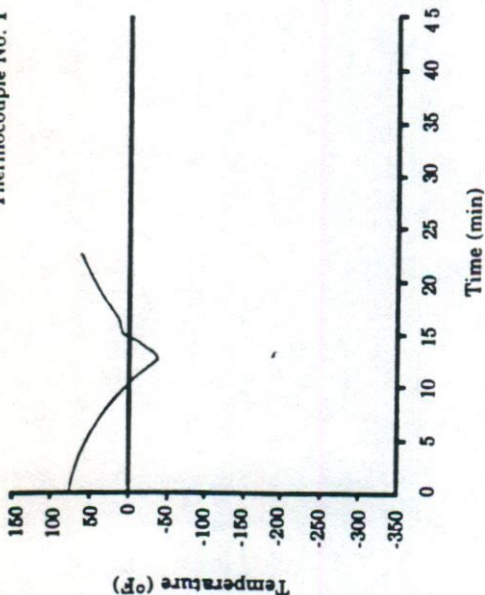
Thermocouple No. 2



Thermocouple No. 4



Thermocouple No. 1



Thermocouple No. 3

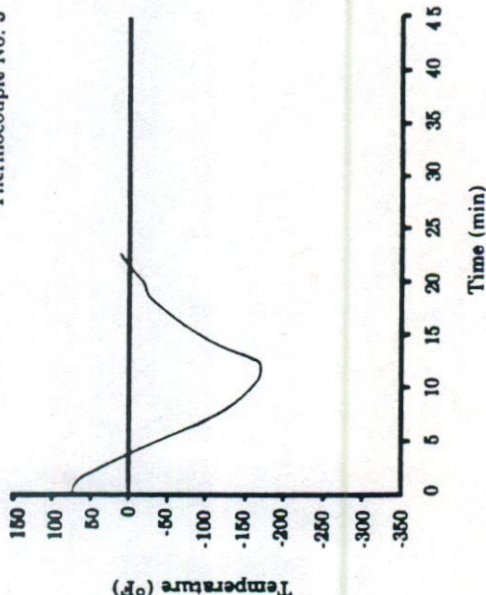
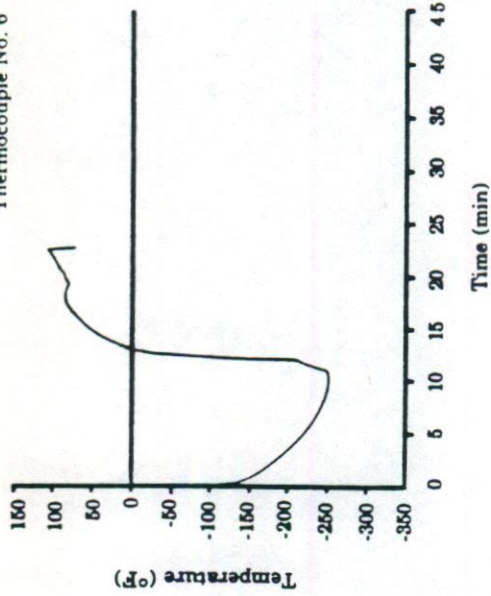


Figure A-1. Temperature Versus Time - Test No. 1

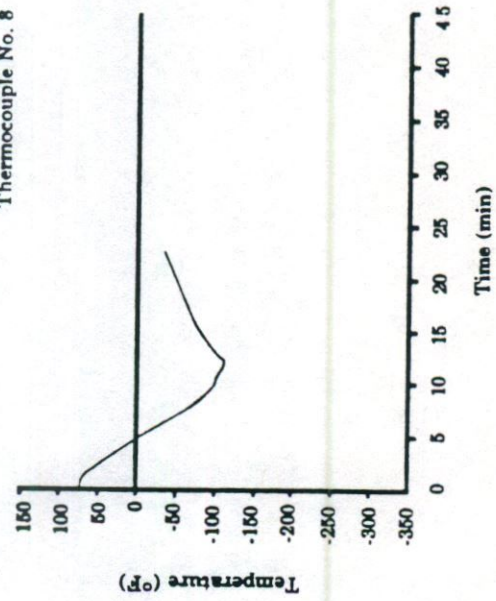
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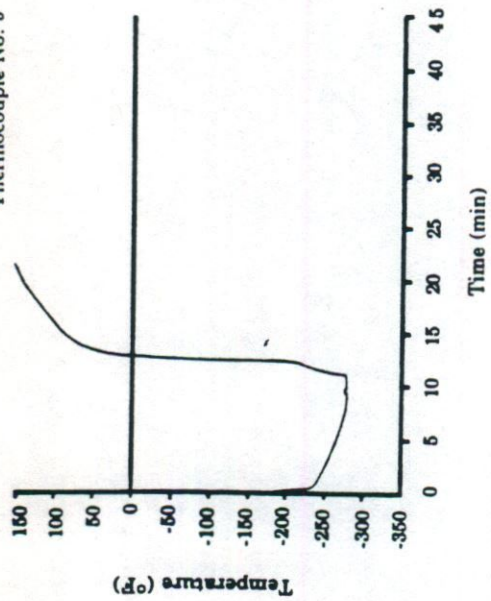
Thermocouple No. 6



Thermocouple No. 8



Thermocouple No. 5



Thermocouple No. 7

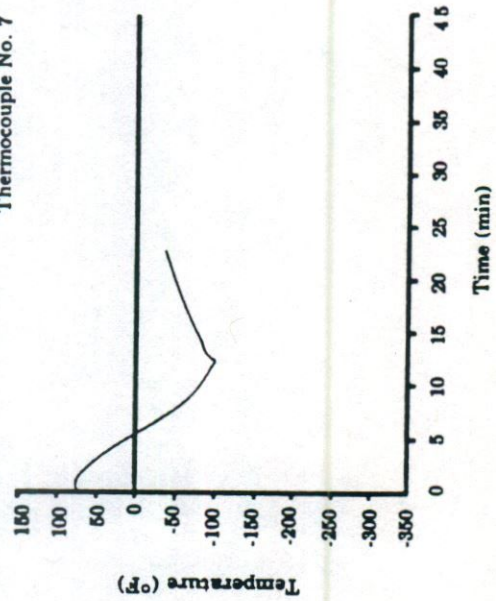


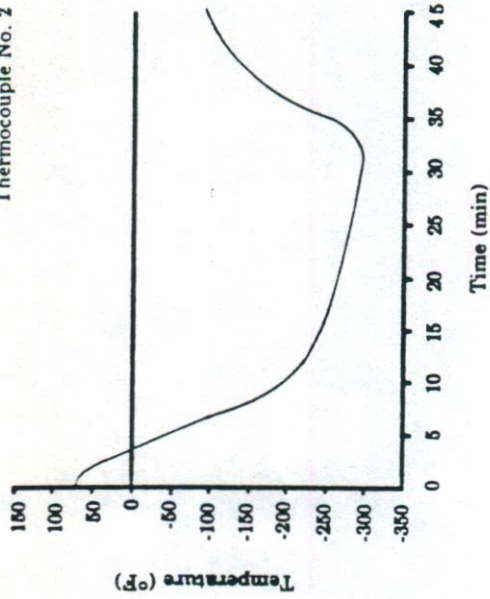
Figure A-1. Temperature Versus Time - Test No. 1 (Cont'd.)

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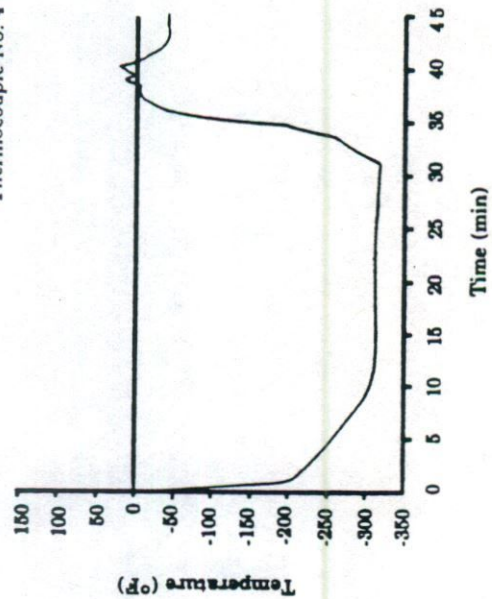


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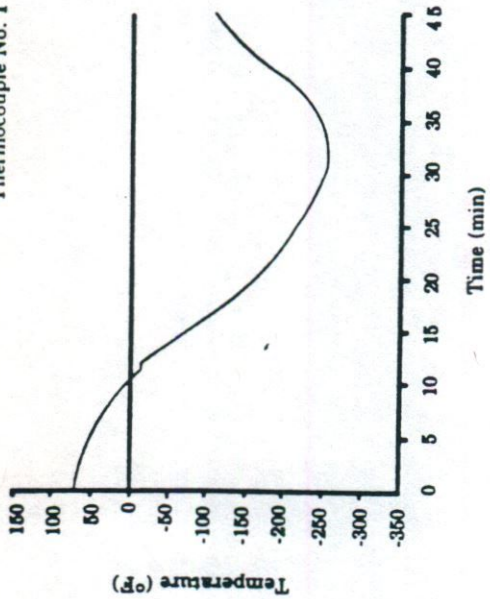
Thermocouple No. 2



Thermocouple No. 4



Thermocouple No. 1



Thermocouple No. 3

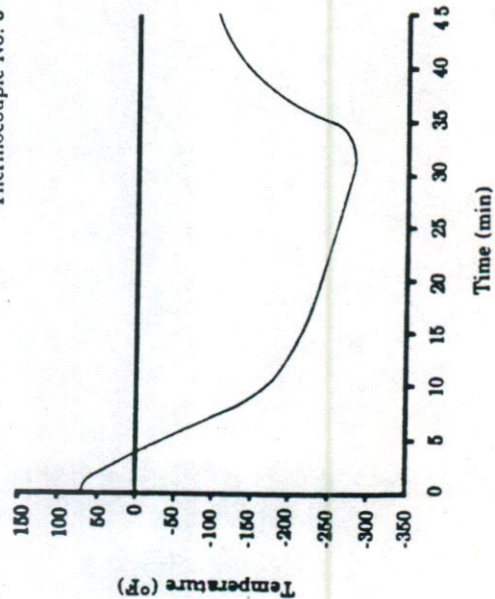
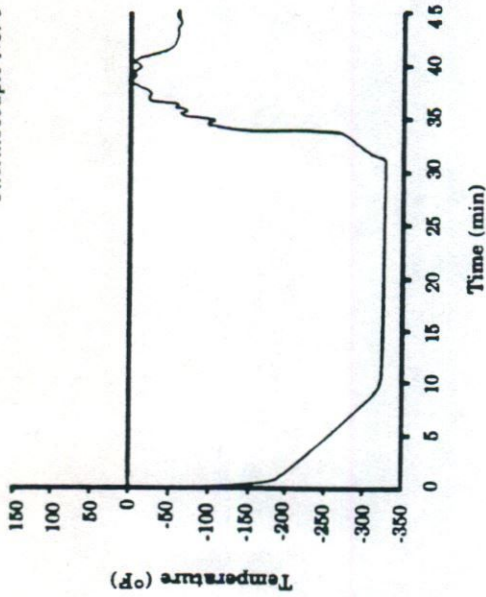


Figure A-2. Temperature Versus Time - Test No. 2

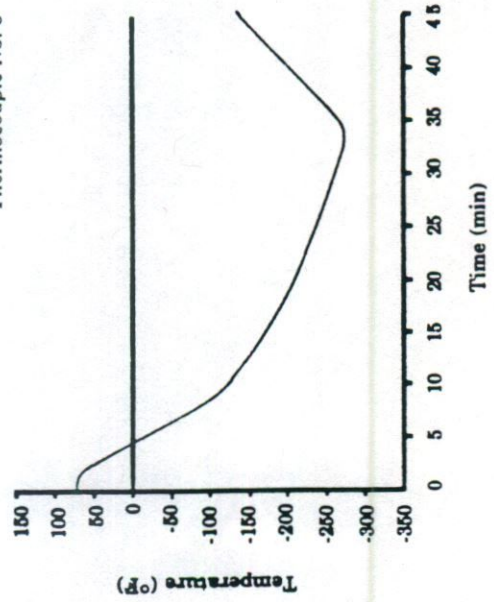
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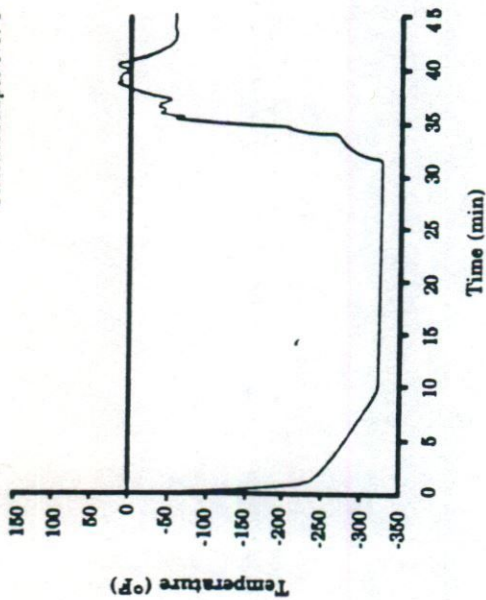
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Thermocouple No. 8



Thermocouple No. 5



Thermocouple No. 7

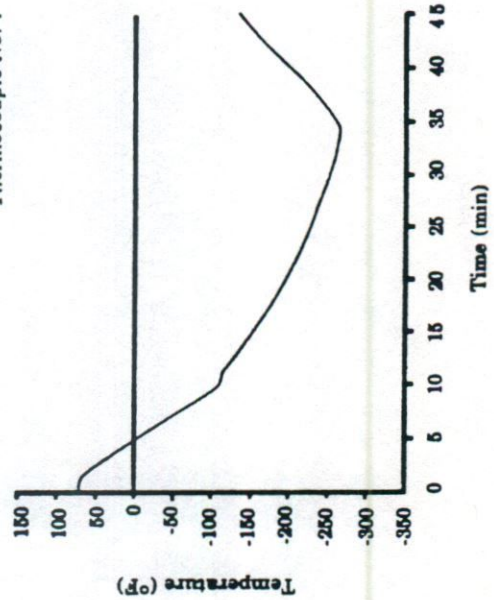


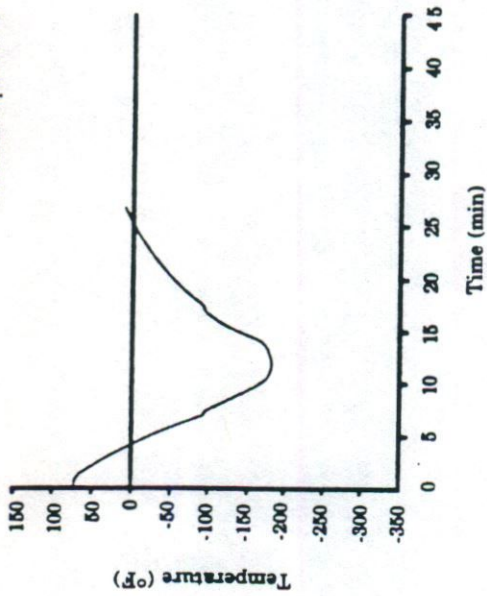
Figure A-2. Temperature Versus Time - Test No. 2 (Cont'd.)

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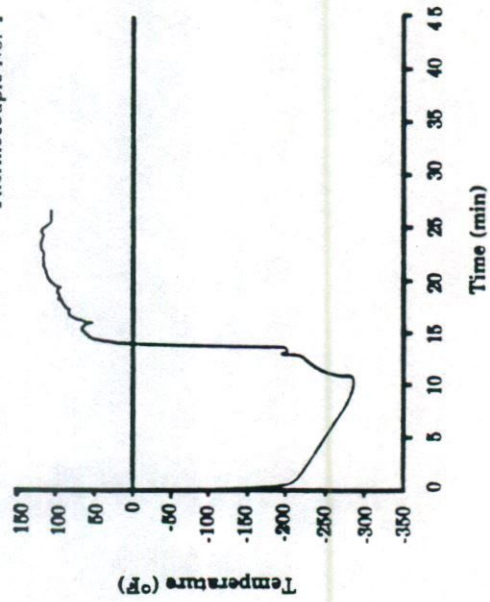


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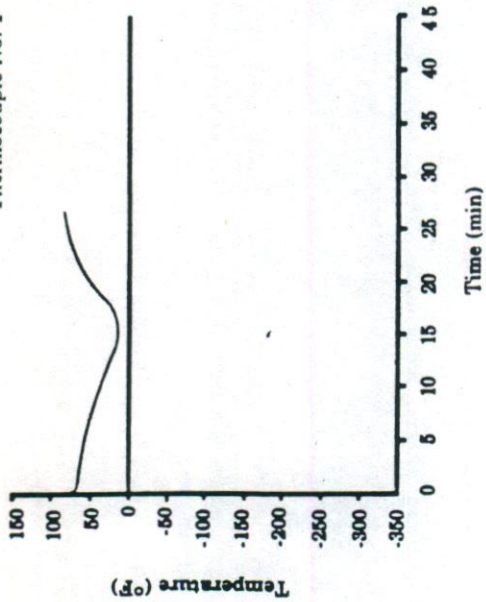
Thermocouple No. 2



Thermocouple No. 4



Thermocouple No. 1



Thermocouple No. 3

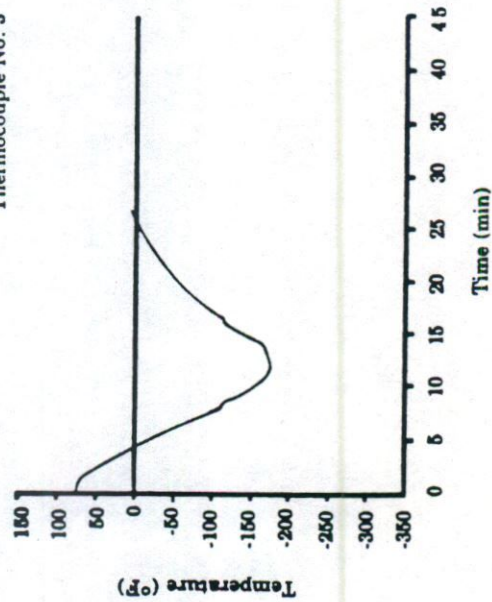


Figure A-3. Temperature Versus Time - Test No. 3

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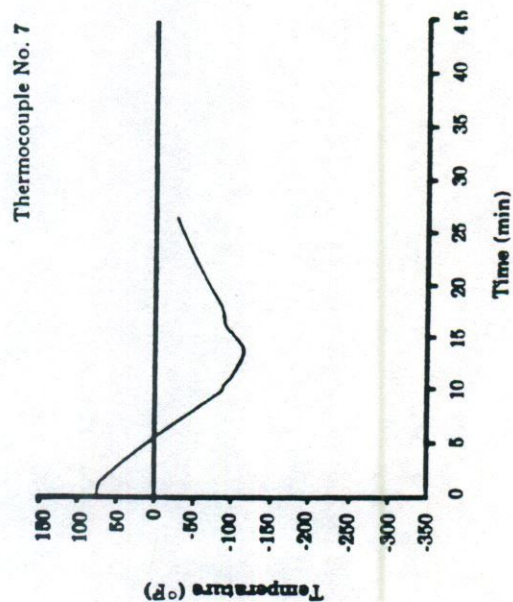
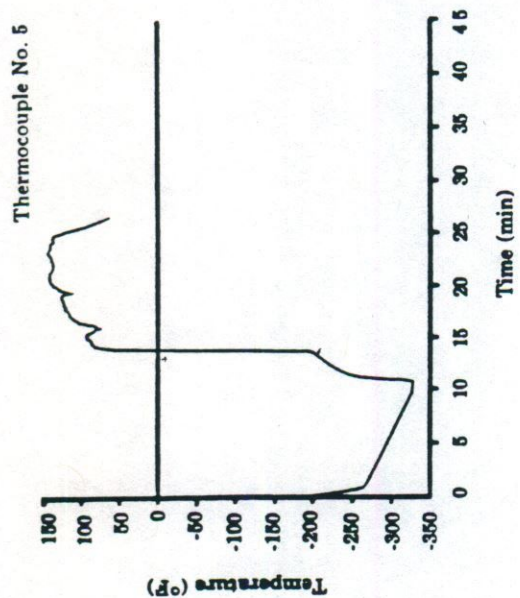
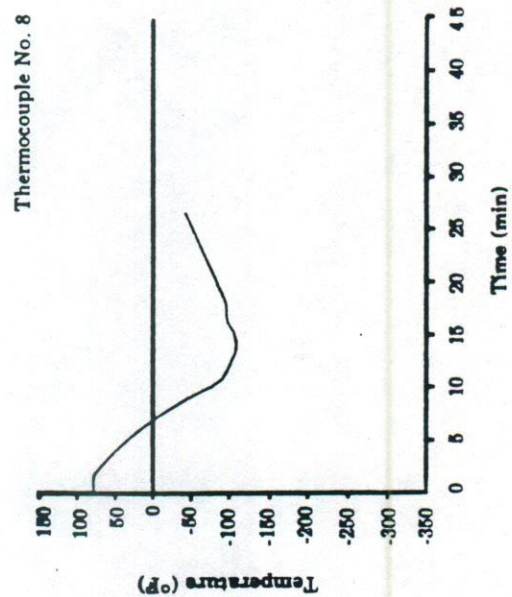
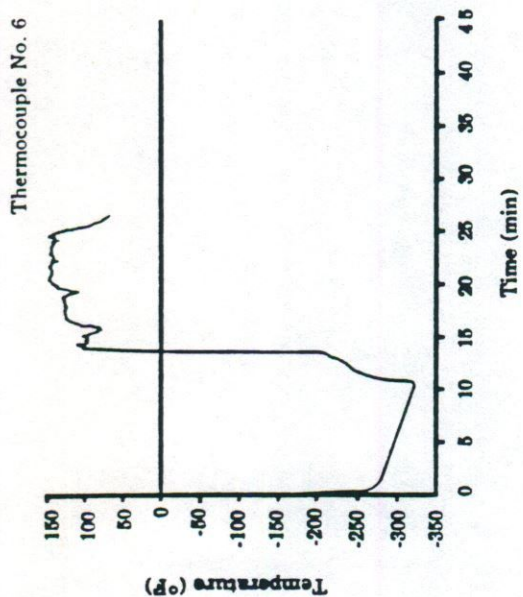


Figure A-3. Temperature Versus Time - Test No. 3 (Cont'd.)

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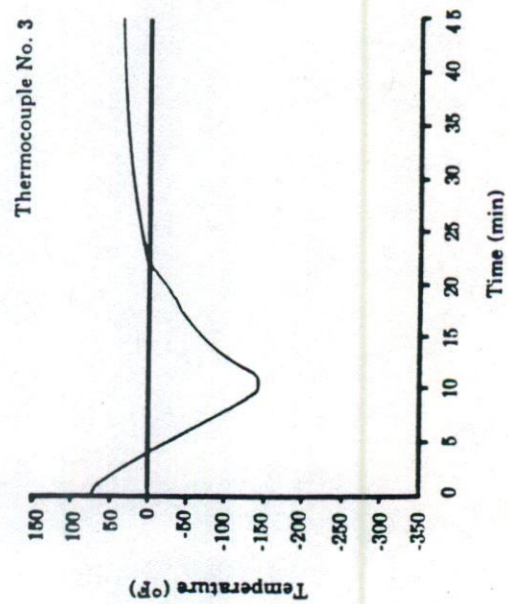
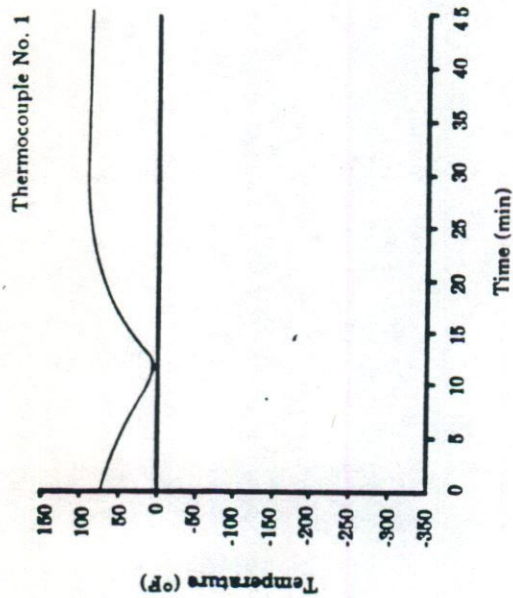
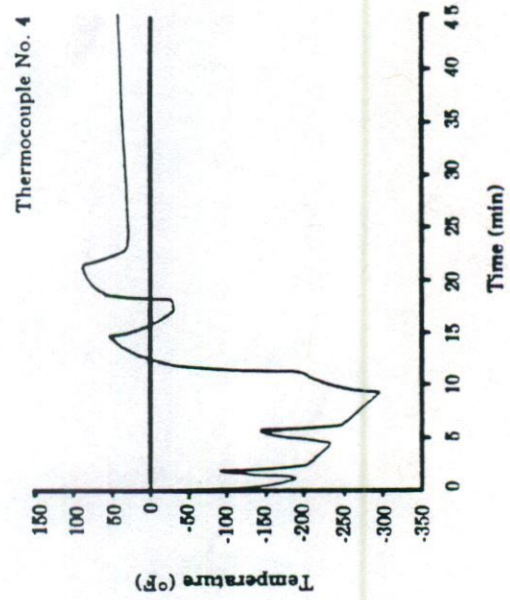
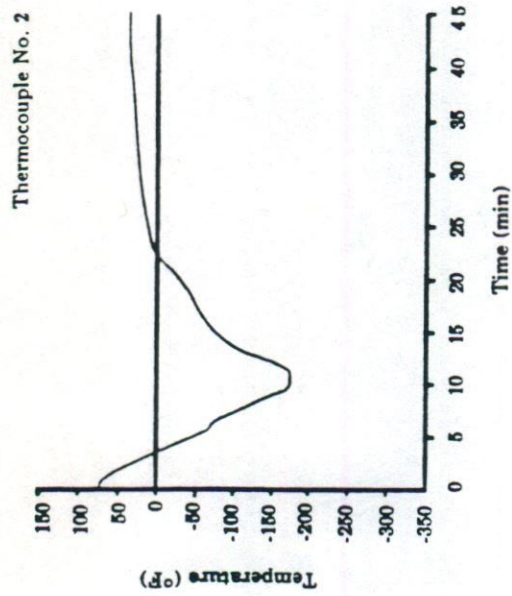


Figure A-4. Temperature Versus Time - Test No. 4

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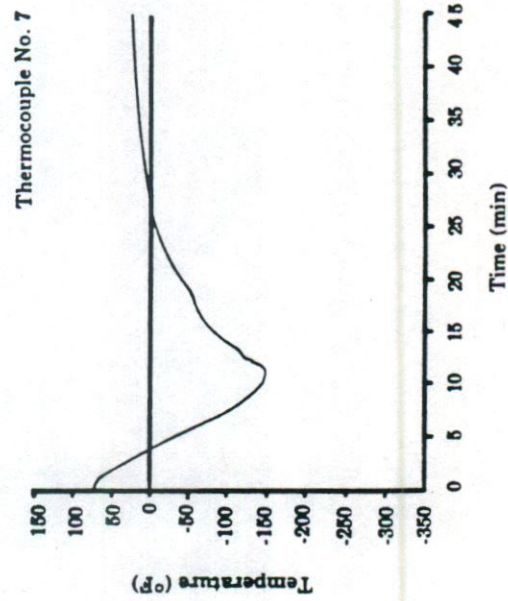
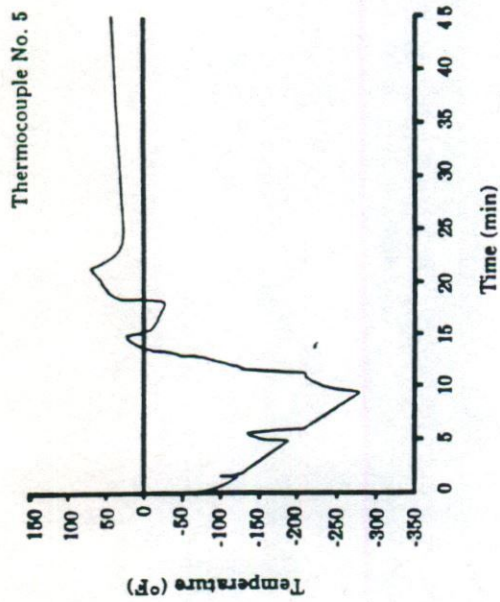
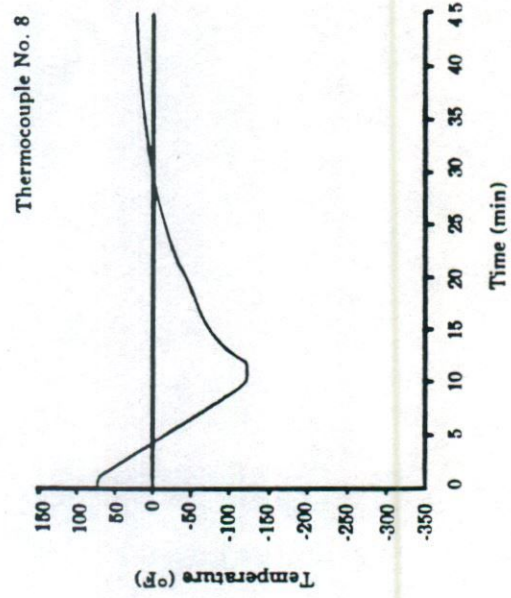
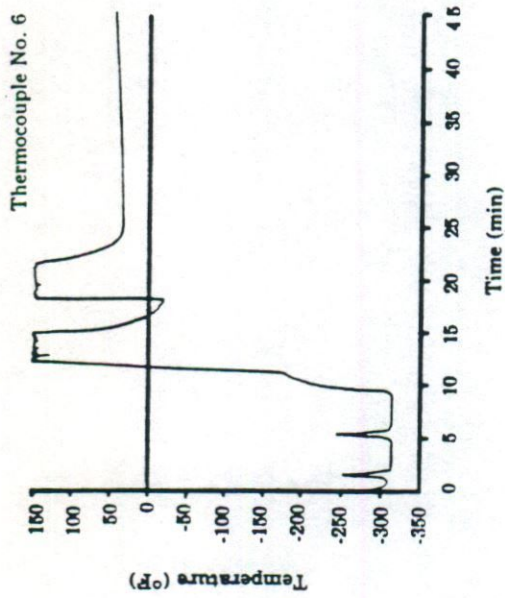


Figure A-4. Temperature Versus Time - Test No. 4 (Cont'd.)

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## APPENDIX B

### Unit No. 8 Temperatures for Stress Analysis

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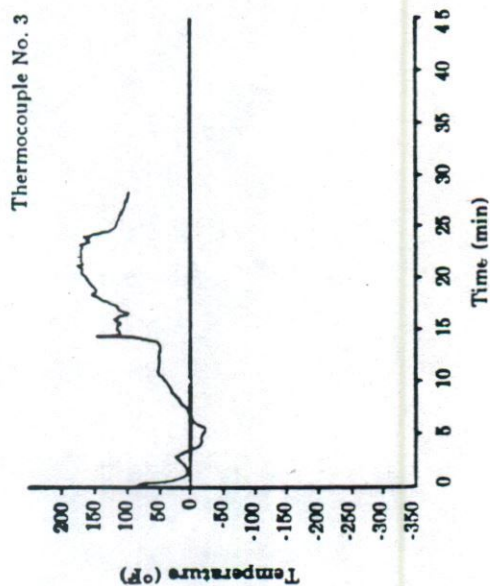
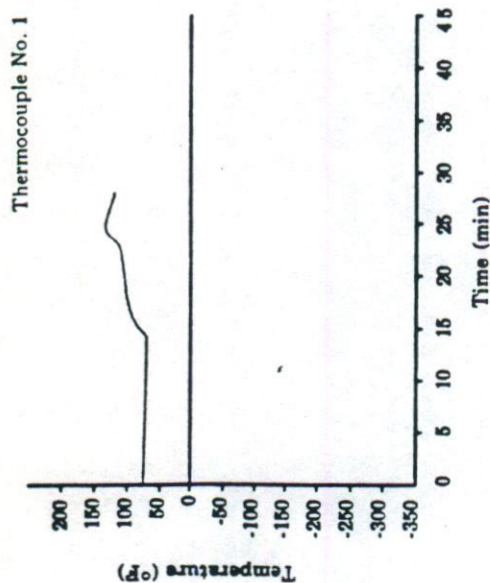
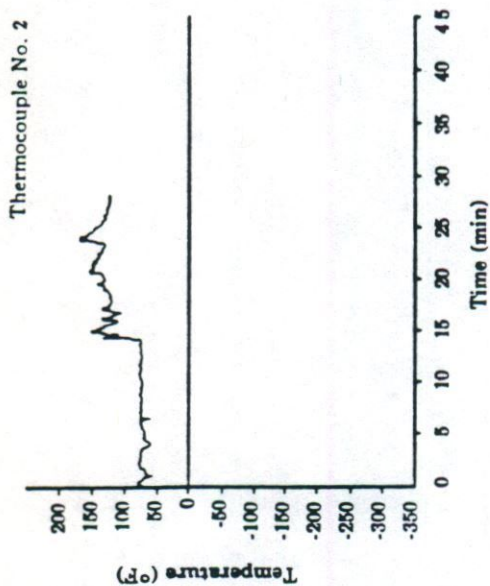


Figure B-1. Temperature Versus Time

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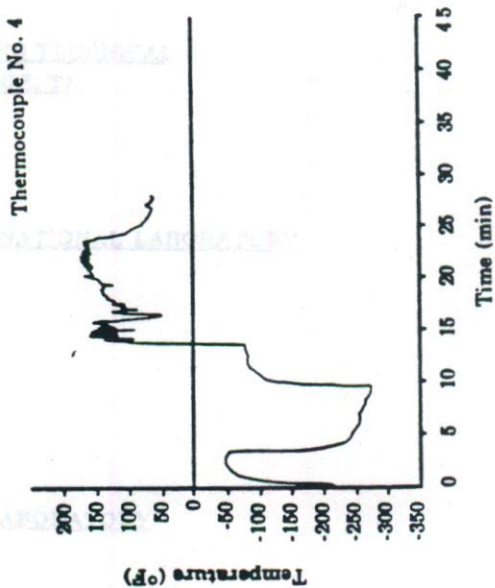
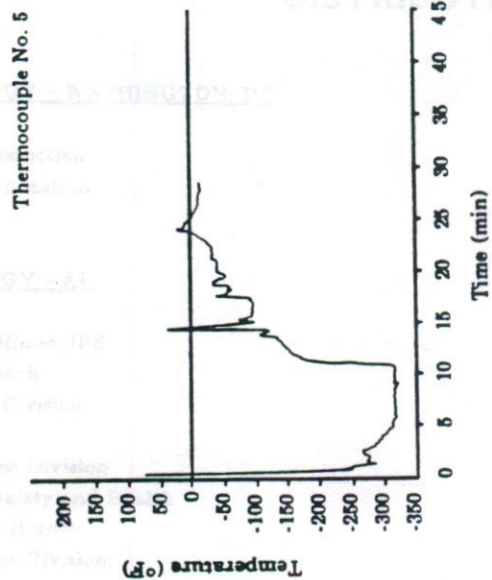


Figure B-1. Temperature Versus Time (Cont'd.)

B-2

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C-1

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